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Search Results - Record(s) 1 through 10 of 10 returned.

☐ 1. Document ID: US 6851561 B2

L10: Entry 1 of 10

File: USPT

Feb 8, 2005

US-PAT-NO: 6851561

DOCUMENT-IDENTIFIER: US 6851561 B2

TITLE: Positively charged membrane

DATE-ISSUED: February 8, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wu; Xiaosong	Pensacola	FL		
Hou; Chung-Jen	Pensacola	FL		
Dharia; Jayesh	Pensacola	FL		
Konstantin; Peter	Boulder	CO		
Yang; Yujing	Newton	MA		

US-CL-CURRENT: 210/490; 210/435, 210/488, 210/493.1, 210/497.01, 210/500.21,
210/500.37, 210/500.38

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWOC	Draw D
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☐ 2. Document ID: US 6780327 B1

L10: Entry 2 of 10

File: USPT

Aug 24, 2004

US-PAT-NO: 6780327

DOCUMENT-IDENTIFIER: US 6780327 B1

TITLE: Positively charged membrane

DATE-ISSUED: August 24, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wu; Xiaosong	Pensacola	FL		
Hou; Chung-Jen	Pensacola	FL		
Dharia; Jayesh	Pensacola	FL		
Konstantin; Peter	Heinade			DE

Yang; Yujing

Pensacola

FL

US-CL-CURRENT: 210/660; 210/488, 210/490, 210/500.21, 210/500.37, 210/500.38,
210/502.1, 427/245, 521/27

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KMOC	Draw Dg
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☐ 3. Document ID: US 6340588 B1

L10: Entry 3 of 10

File: USPT

Jan 22, 2002

US-PAT-NO: 6340588

DOCUMENT-IDENTIFIER: US 6340588 B1

TITLE: Matrices with memories

DATE-ISSUED: January 22, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Nova; Michael P.	Rancho Santa Fe	CA		
Potash; Hanan	Austin	TX		

US-CL-CURRENT: 435/287.1; 435/287.2, 435/288.1, 435/288.3, 435/288.4, 435/288.7,
530/300, 530/334, 530/350, 536/23.1, 536/24.3, 536/25.3

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KMOC	Draw Dg
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☐ 4. Document ID: US 5049282 A

L10: Entry 4 of 10

File: USPT

Sep 17, 1991

US-PAT-NO: 5049282

DOCUMENT-IDENTIFIER: US 5049282 A

TITLE: Semipermeable composite membranes

DATE-ISSUED: September 17, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KMOC	Draw Dg
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☐ 5. Document ID: US 5039421 A

L10: Entry 5 of 10

File: USPT

Aug 13, 1991

US-PAT-NO: 5039421

DOCUMENT-IDENTIFIER: US 5039421 A

TITLE: Solvent stable membranes

DATE-ISSUED: August 13, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Perry; Mordechai	Petach Tikva			IL
Nemas; Mara	Neve Monosson			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.43, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWAC	Draw D
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☐ 6. Document ID: US 5032282 A

L10: Entry 6 of 10

File: USPT

Jul 16, 1991

US-PAT-NO: 5032282

DOCUMENT-IDENTIFIER: US 5032282 A

TITLE: Solvent-stable semipermeable composite membranes

DATE-ISSUED: July 16, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monosson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.23, 210/500.43, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWAC	Draw D
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☐ 7. Document ID: US 5024765 A

L10: Entry 7 of 10

File: USPT

Jun 18, 1991

US-PAT-NO: 5024765

DOCUMENT-IDENTIFIER: US 5024765 A

TITLE: Composite membranes and processes using them

DATE-ISSUED: June 18, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Perry; Mordechai	Petach Tikva			IL
Nemas; Mara	Neve Monosson			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.41, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWAC	Draw D
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☐ 8. Document ID: US 4833014 A

L10: Entry 8 of 10

File: USPT

May 23, 1989

US-PAT-NO: 4833014

DOCUMENT-IDENTIFIER: US 4833014 A

TITLE: Composite membranes useful for the separation of organic compounds of low molecular weight from aqueous inorganic salts containing solutions

DATE-ISSUED: May 23, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach-Tikva			IL
Kotraro; Reuven	Rishon-Letzion			IL

US-CL-CURRENT: 428/308.4; 427/333, 427/340, 427/393.5, 427/412.1, 428/310.5,
428/315.7, 428/315.9, 428/316.6, 428/336

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWAC	Draw D
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☐ 9. Document ID: US 4778596 A

L10: Entry 9 of 10

File: USPT

Oct 18, 1988

US-PAT-NO: 4778596

DOCUMENT-IDENTIFIER: US 4778596 A

TITLE: Semipermeable encapsulated membranes, process for their manufacture and their use

DATE-ISSUED: October 18, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Aviv; Gershon	Tel Aviv			IL
Perry; Mordechai	Petach Tikvah			IL
Kotraro; Reuven	Rehovot			IL

US-CL-CURRENT: 210/638; 210/490, 210/500.38, 210/651, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw Dg
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☐ 10. Document ID: US 4767645 A

L10: Entry 10 of 10

File: USPT

Aug 30, 1988

US-PAT-NO: 4767645

DOCUMENT-IDENTIFIER: US 4767645 A

TITLE: Composite membranes useful for the separation of organic compounds of low molecular weight from aqueous inorganic salts containing solutions

DATE-ISSUED: August 30, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach-Tikva			IL
Kotraro; Reuven	Rishon-Letzion			IL

US-CL-CURRENT: 427/386; 427/385.5, 427/393.5, 427/412.1, 427/412.3, 427/412.4, 427/412.5

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw Dg
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L9 and covalent bond

10

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Search Results - Record(s) 1 through 10 of 10 returned.

☐ 1. Document ID: US 6851561 B2

L10: Entry 1 of 10

File: USPT

Feb 8, 2005

US-PAT-NO: 6851561

DOCUMENT-IDENTIFIER: US 6851561 B2

TITLE: Positively charged membrane

DATE-ISSUED: February 8, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wu; Xiaosong	Pensacola	FL		
Hou; Chung-Jen	Pensacola	FL		
Dharia; Jayesh	Pensacola	FL		
Konstantin; Peter	Boulder	CO		
Yang; Yujing	Newton	MA		

US-CL-CURRENT: 210/490; 210/435, 210/488, 210/493.1, 210/497.01, 210/500.21,
210/500.37, 210/500.38

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KNOC	Draw Ds
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☐ 2. Document ID: US 6780327 B1

L10: Entry 2 of 10

File: USPT

Aug 24, 2004

US-PAT-NO: 6780327

DOCUMENT-IDENTIFIER: US 6780327 B1

TITLE: Positively charged membrane

DATE-ISSUED: August 24, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wu; Xiaosong	Pensacola	FL		
Hou; Chung-Jen	Pensacola	FL		
Dharia; Jayesh	Pensacola	FL		
Konstantin; Peter	Heinade			DE

Yang; Yujing

Pensacola

FL

US-CL-CURRENT: 210/660; 210/488, 210/490, 210/500.21, 210/500.37, 210/500.38,
210/502.1, 427/245, 521/27

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KNOC	Draw D
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☐ 3. Document ID: US 6340588 B1

L10: Entry 3 of 10

File: USPT

Jan 22, 2002

US-PAT-NO: 6340588

DOCUMENT-IDENTIFIER: US 6340588 B1

TITLE: Matrices with memories

DATE-ISSUED: January 22, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Nova; Michael P.	Rancho Santa Fe	CA		
Potash; Hanan	Austin	TX		

US-CL-CURRENT: 435/287.1; 435/287.2, 435/288.1, 435/288.3, 435/288.4, 435/288.7,
530/300, 530/334, 530/350, 536/23.1, 536/24.3, 536/25.3

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KNOC	Draw D
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☐ 4. Document ID: US 5049282 A

L10: Entry 4 of 10

File: USPT

Sep 17, 1991

US-PAT-NO: 5049282

DOCUMENT-IDENTIFIER: US 5049282 A

TITLE: Semipermeable composite membranes

DATE-ISSUED: September 17, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KNOC	Draw D
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☐ 5. Document ID: US 5039421 A

L10: Entry 5 of 10

File: USPT

Aug 13, 1991

US-PAT-NO: 5039421

DOCUMENT-IDENTIFIER: US 5039421 A

TITLE: Solvent stable membranes

DATE-ISSUED: August 13, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Perry; Mordechai	Petach Tikva			IL
Nemas; Mara	Neve Monosson			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.43, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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☐ 6. Document ID: US 5032282 A

L10: Entry 6 of 10

File: USPT

Jul 16, 1991

US-PAT-NO: 5032282

DOCUMENT-IDENTIFIER: US 5032282 A

TITLE: Solvent-stable semipermeable composite membranes

DATE-ISSUED: July 16, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monosson			IL
Perry; Mordechai	Petach Tikva			IL
Ketraro; Reuven	Rishon Letzion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.23, 210/500.43, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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☐ 7. Document ID: US 5024765 A

L10: Entry 7 of 10

File: USPT

Jun 18, 1991

US-PAT-NO: 5024765

DOCUMENT-IDENTIFIER: US 5024765 A

TITLE: Composite membranes and processes using them

DATE-ISSUED: June 18, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Perry; Mordechai	Petach Tikva			IL
Nemas; Mara	Neve Monosson			IL
Katraro; Reuven	Rishon Lezion			IL

US-CL-CURRENT: 210/651; 210/490, 210/500.41, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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☐ 8. Document ID: US 4833014 A

L10: Entry 8 of 10

File: USPT

May 23, 1989

US-PAT-NO: 4833014

DOCUMENT-IDENTIFIER: US 4833014 A

TITLE: Composite membranes useful for the separation of organic compounds of low molecular weight from aqueous inorganic salts containing solutions

DATE-ISSUED: May 23, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach-Tikva			IL
Kotraro; Reuven	Rishon-Letzion			IL

US-CL-CURRENT: 428/308.4; 427/333, 427/340, 427/393.5, 427/412.1, 428/310.5, 428/315.7, 428/315.9, 428/316.6, 428/336

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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☐ 9. Document ID: US 4778596 A

L10: Entry 9 of 10

File: USPT

Oct 18, 1988

US-PAT-NO: 4778596

DOCUMENT-IDENTIFIER: US 4778596 A

TITLE: Semipermeable encapsulated membranes, process for their manufacture and their use

DATE-ISSUED: October 18, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Aviv; Gershon	Tel Aviv			IL
Perry; Mordechai	Petach Tikvah			IL
Kotraro; Reuven	Rehovot			IL

US-CL-CURRENT: 210/638; 210/490, 210/500.38, 210/651, 210/654

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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☐ 10. Document ID: US 4767645 A

L10: Entry 10 of 10

File: USPT

Aug 30, 1988

US-PAT-NO: 4767645

DOCUMENT-IDENTIFIER: US 4767645 A

TITLE: Composite membranes useful for the separation of organic compounds of low molecular weight from aqueous inorganic salts containing solutions

DATE-ISSUED: August 30, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Linder; Charles	Rehovot			IL
Nemas; Mara	Neve Monoson			IL
Perry; Mordechai	Petach-Tikva			IL
Kotraro; Reuven	Rishon-Letzion			IL

US-CL-CURRENT: 427/386; 427/385.5, 427/393.5, 427/412.1, 427/412.3, 427/412.4, 427/412.5

Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	KWIC	Draw D
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L9 and covalent bond

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	IBM Technical Disclosure Bulletins

Term:	L9 and covalent bond
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Display:	<input type="text" value="10"/> Documents in Display Format: <input type="text" value="CIT"/> Starting with Number <input type="text" value="1"/>
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Search History

DATE: Tuesday, July 05, 2005 [Printable Copy](#) [Create Case](#)

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DB=USPT; PLUR=YES; OP=ADJ

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result set

<u>L10</u>	L9 and covalent bond	10	<u>L10</u>
<u>L9</u>	l3 and coating same amino	27	<u>L9</u>
<u>L8</u>	L7 and l6	1	<u>L8</u>
<u>L7</u>	264/49.ccls.	610	<u>L7</u>
<u>L6</u>	l3 and amino group	94	<u>L6</u>
<u>L5</u>	l3 and amono group	0	<u>L5</u>
<u>L4</u>	L3 and posttreatment same amine	0	<u>L4</u>
<u>L3</u>	L2 and amine	330	<u>L3</u>
<u>L2</u>	polyimide same membrane and pores	900	<u>L2</u>
<u>L1</u>	polimide membrane and support and asymmetric	0	<u>L1</u>

END OF SEARCH HISTORY

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
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L2	2	("4664669").PN.	USPAT; EPO; DERWENT	OR	OFF	2005/07/05 12:54
L3	0	"utem 1000" and polyimide	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:55
L4	0	"utem 1000"	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:55
L5	451	"ultem 1000"	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:55
L6	0	15 nad polyimide and amino	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:56
L7	79	15 and polyimide and amino	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:56
L8	45	17 and coating	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:57
L9	0	18 and coating with amine	USPAT; EPO; DERWENT	AND	ON	2005/07/05 12:57
L10	10	18 and coating with amino	USPAT; EPO; DERWENT	AND	ON	2005/07/05 13:02
L11	0	110 and covalent bonding	USPAT; EPO; DERWENT	AND	ON	2005/07/05 13:02
L12	2	18 and covalent	USPAT; EPO; DERWENT	AND	ON	2005/07/05 13:03

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BRS: BRS: Pending Active

L1: (2) ("4798847").PN.
 L2: (2) ("4664669").PN.
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 L4: (0) "utem 1000"
 L5: (451) "ultem 1000"
 L6: (0) l5 nad polyimide and amin
 L7: (79) l5 and polyimide and ami
 L8: (45) l7 and coating
 L9: (0) l8 and coating with amine
 L10: (10) l8 and coating with amin
 L11: (0) l10 and covalent bonding
 L12: (2) l8 and covalent
 L13: (15) functionalized same poly
 L14: (0) l13 and amina
 L15: (2) primary amine and l13
 L16: (242) 210/500.39
 L17: (68) l16 and primary amine
 L18: (42) l17 and coating coating

Failed

USPAT: EPO: DERWENT

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l17 and coating coating

BRS form ISIR form Image Text HTML

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2	<input type="checkbox"/>	<input type="checkbox"/>	US 6544419 B1	20030408	12	Method of preparing a composite polymer and silica-based membrane	210/500.26	210/500.21;
3	<input type="checkbox"/>	<input type="checkbox"/>	US 6544418 B1	20030408	13	Preparing and regenerating a composite polymer and silica-based	210/500.26	210/500.37;
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5	<input type="checkbox"/>	<input type="checkbox"/>	US 6306301 B1	20011023	12	Silica-based membrane sorbent for heavy metal sequestration	210/500.26	210/500.37;
6	<input type="checkbox"/>	<input type="checkbox"/>	US 6260715 B1	20010717	36	Means for the biological purification of a biological fluid	210/490	210/500.43;
7	<input type="checkbox"/>	<input type="checkbox"/>	US 6113794 A	20000905	6	Composite solvent resistant nanofiltration membranes	210/650	422/101;
8	<input type="checkbox"/>	<input type="checkbox"/>	US 6063278 A	20000516	8	Composite polyamide reverse osmosis membrane and method of pr	210/500.38	210/500.27;
9	<input type="checkbox"/>	<input type="checkbox"/>	US 6015495 A	20000118	9	Composite polyamide reverse osmosis membrane and method of pr	210/500.38	210/490;
10	<input type="checkbox"/>	<input type="checkbox"/>	US 5922203 A	19990713	11	Amine monomers and their use in preparing interfacially synthesized me	210/500.37	264/41;
11	<input type="checkbox"/>	<input type="checkbox"/>	US 5753008 A	19980519	10	Solvent resistant hollow fiber vapor permeation membranes and modules	95/45	210/500.38;

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L24: Entry 3 of 5

File: USPT

Apr 28, 1998

DOCUMENT-IDENTIFIER: US 5744039 A

TITLE: Composite semipermeable membrane and production method thereof

Abstract Text (1):

Disclosed are a composite semipermeable membrane which is excellent in the chlorine resistance and also gives a high desalting faculty and a large water flux, in particular, under a low operation pressure and a production method thereof. The composite semipermeable membrane comprises a porous base material having formed thereon a semipermeable thin film formed by crosslinking polymerization of 2,6-diaminotoluene monomer or a mixture of 2,6-diaminotoluene monomer and other amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with the amino group(s) of the monomer(s) in one molecule in the presence of an amine salt.

Brief Summary Text (2):

The present invention relates to a composite semipermeable membrane for selectively separating a mixture of various kinds of liquids and a production method thereof, and more specifically to a composite semipermeable membrane composed of a porous base material having formed thereon a semipermeable thin membrane formed by conducting crosslinking polymerization of 2,6-diaminotoluene monomer or a mixture of the monomer and other amino compound monomer with an amine salt in the presence of a polyfunctional crosslinking agent, and to a production method thereof.

Brief Summary Text (13):

That is, according to one aspect of the present invention, there is provided a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable film formed by conducting crosslinking polymerization of 2,6-diaminotoluene monomer or a mixture of 2,6-diaminotoluene monomer and other amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with the amino group(s) of the foregoing monomer(s) in one molecule in the presence of an amine salt. Also, as a preferred constitution, the composite semipermeable membrane has a faculty of a salt rejection of at least 80% to an aqueous solution of 1500 ppm of sodium chloride in the operation under the condition of 25.degree. C. and an operation pressure of 5 kg/cm.sup.2 together with a faculty of water flux of at least 0.4 m.sup.3 /m.sup.2 per day.

Brief Summary Text (14):

According to another object of the present invention, there is provided a production method of a composite semipermeable membrane, which comprises coating or impregnating a porous base material with a solution containing 2,6-diaminotoluene monomer or a mixture thereof with other amino compound monomer and an amine salt and thereafter contacting the coated or impregnated layer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with the amino group(s) in the monomer(s) in one molecule to form a semipermeable thin film on the porous base material by causing a crosslinking polymerization. Also, as a preferred constitution, the composite semipermeable membrane obtained by the production method of the present invention has a faculty of a salt rejection of at least 80% to an aqueous solution of 1500 ppm of sodium chloride in the operation under the condition of 25.degree. C. and an operation pressure of 5 kg/cm.sup.2

together with a faculty of a water flux of at least 0.4 m.sup.3 /m.sup.2 per day.

Brief Summary Text (16):

There is no particular restriction on the porous base material being used in this invention if the base material can support thereon a thin film but usually a ultrafiltration film having fine pores of from 1 to 50 nm on the surface thereof is used. Examples of the material being used as the porous base material are polysulfone, polyether sulfone, polyacrylonitrile, polyimide, polyamide, an ethylene-vinyl alcohol copolymer, and cellulose acetate but from the points of the heat resistance, the chlorine resistance, etc., polysulfone and polyether sulfone are preferred. Also, such a porous film may be reinforced by backing with a woven fabric or nonwoven fabric.

Brief Summary Text (18):

As other amino compound monomer than 2,6-diaminotoluene monomer being used in the present invention, an aliphatic, alicyclic, aromatic or heteroaromatic compound having at least two primary amino groups and/or secondary amino groups in one molecule and having a solubility of at least 0.05% by weight to water, an aliphatic alcohol having from 1 to 4 carbon atoms, or a mixed solvent thereof at room temperature is desirable. Practical examples of the preferred amino compound monomer are m-phenylenediamine, N,N'-dimethyl-m-phenylenediamine, 2,4-diaminopyridine, 2,4,6-trimethyl-m-phenylenediamine, 4,4-diaminophenylsulfone, piperazine, and aminopiperidine and in these compounds, m-phenylenediamine is more preferably used.

Brief Summary Text (19):

These amino compound monomers each may have a functional group such as a methoxy group, a chlorine atom, a carboxyl group, etc., and they can be used singly or as a mixture of them.

Brief Summary Text (20):

As to the mixing ratio of the other amino compound monomer and 2,6-diaminotoluene, it is preferred that the content of the other amino compound monomer is not more than 60% by weight to the content of 2,6-diaminotoluene monomer, i.e., that the other amino compound is used in an amount corresponding to 60% by weight or less of the content of 2,6-diaminotoluene monomer. If the content of the other amino compound monomer is over 60% by weight, the influence of the other amino compound monomer to the membrane faculty becomes dominant, whereby the feature of using 2,6-diaminotoluene giving a high water flux even at a very low operation pressure is sometimes restrained. For example, in the case that the other amino compound monomer is m-phenylenediamine, as will be described later in Comparative Examples 6 to 8, when the content of m-phenylenediamine is over 60% by weight to the content of 2,6-diaminotoluene, it sometimes happens that the water flux of the membrane faculty is reduced. Accordingly, in order that the composite semipermeable membrane has a high water flux even under a very low operation pressure of 5 kg/cm.sup.2, the mixing ratio of the other amino compound monomer is preferably not more than 60% by weight, and more preferably not more than 50% by weight to the amount of 2,6-diaminotoluene.

Brief Summary Text (24):

The polyfunctional crosslinking agent being used in the present invention is a compound having at least two functional groups capable of reacting the amino group of 2,6-diaminotoluene or the amino groups of 2,6-diaminotoluene and the foregoing other amino compound monomer in one molecule and specific examples of the functional group are an acid halide group, an acid anhydride group, an isocyanate group, a halogensulfonyl group, a haloformate group, and an N-haloformyl group.

Brief Summary Text (26):

In the present invention, from the point of increasing the rejection faculty of the membrane, the compound having at least two acid halide groups in one molecule, such

as isophthaloyl chloride, trimesic acid chloride, cyclopentanetetracarboxylic acid chloride, etc., is preferably used and trimesic acid chloride is more preferably used.

Brief Summary Text (27):

In the case of producing the composite semipermeable membrane of the present invention, 2,6-diaminotoluene monomer or a mixture of 2,6-diaminotoluene monomer and the foregoing other amino compound monomer are crosslinking polymerized with the foregoing polyfunctional crosslinking agent in the presence of the foregoing amine salt on the porous base material to form a semipermeable super-thin film. The thickness of the semipermeable thin film is preferably from 100 to 500 nm. The semipermeable thin film is formed by coating or impregnating the porous base material with a solution containing 2,6-diaminotoluene monomer or a mixture of 2,6-diaminotoluene, the foregoing other amino compound monomer, and the foregoing amine salt (hereinafter, the solution is referred to as base liquid) and then contacting the coated or impregnated layer with the foregoing polyfunctional crosslinking agent to cause a crosslinking polymerization. The contacting can be conducted by conventional methods such as an immersing method and a spraying method. In addition, at the crosslinking polymerization, it is preferred to heat the system for completing the crosslinkage.

Brief Summary Text (32):

Also, in the crosslinking polymerization, for more sufficiently carrying out crosslinking, that is, for completing the crosslinking polymerization by the polyfunctional crosslinking agent and 2,6-diaminotoluene or a mixture of 2,6-diaminotoluene and the foregoing other amino compound monomer, it is preferred to carry out a heat treatment. Usually, the heating temperature is from 60.degree. C. to 160.degree. C. and the heating time is usually from about 1 minute to 30 minutes.

Detailed Description Text (11):

By following the same procedure as Example 1 except that the mixing ratio of the other amino compound monomer was over 60% by weight to the amount of 2,6-diaminotoluene and also the constituting components were changed as shown in Table 1 below, comparative composite semipermeable membranes were prepared and the performances of these membranes are shown in Table 1 below. As shown in Table 1 below, the water flux is less than those of the membrane in Example 1 and the membranes in Examples 13 to 18.

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US Reference Group (2):
3878109 19750400 Ikeda et al. 210/500.38

US Reference Group (3):
3996318 19761200 van Heuven 210/500.38

US Reference Group (8):
4769148 19880900 Fibeger et al. 210/500.38

US Reference Group (11):
5019264 19910500 Arthur 210/500.38

US Reference Group (12):
5160619 19921100 Yamaguchi et al. 210/500.38

US Reference Group (13):
5336409 19940800 Hachisuka et al. 210/500.38

CLAIMS:

1. A liquid separation composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, wherein the interfacial polymerization takes place in the presence of an amine salt.
3. The composite liquid separation semipermeable membrane of claim 1, wherein the mixing ratio of the other amino compound monomer is not more than 60% by weight to the 2,6-diaminotoluene monomer.
4. The composite liquid separation semipermeable membrane of claim 1, wherein the other amino compound monomer is m-phenylenediamine, the amine salt is a salt composed of triethylamine and camphorsulfonic acid, and the polyfunctional crosslinking agent is trimesic acid chloride.
5. A production method of a composite liquid separation semipermeable membrane, which comprises coating or impregnating a porous base material with a solution containing (1) 2,6-diaminotoluene monomer or a mixture of 2,6-diaminotoluene monomer and other amino compound monomer and (2) an amino salt and thereafter, contacting the coated or impregnated layer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with the amino group(s) of the monomer(s) to cause an interfacial crosslinking polymerization, thereby to form a semipermeable thin film on the porous base material.
7. The production method of claim 5, wherein the mixing ratio of the other amino compound monomer is not more than 60% by weight to the 2,6-diaminotoluene monomer.
8. The production method of claim 5, wherein the other amino compound monomer is m-phenylenediamine, the amine salt is a salt composed of triethylamine and camphorsulfonic acid, and the polyfunctional crosslinking agent is trimesic acid chloride.
9. A composite liquid separation semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two

functional groups capable of reacting with amino groups of the monomers, wherein the interfacial polymerization takes place in the presence of an amine salt.

11. The composite liquid separation semipermeable membrane of claim 9, wherein the mixing ratio of the other amino compound monomer is not more than 60% by weight to the 2,6-diaminotoluene monomer.

12. The composite liquid separation semipermeable membrane of claim 9, wherein the other amino compound monomer is m-phenylenediamine, the amine salt is a salt composed of triethylamine and camphorsulfonic acid, and the polyfunctional crosslinking agent is trimesic acid chloride.

13. A liquid separation method, comprising:

applying, to a liquid, a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of:

(a) a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, or

(b) a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomers,

wherein the interfacial polymerization takes place in the presence of an amine salt.

14. A method of producing fresh water, comprising:

applying, to a liquid, a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of:

(a) a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, or

(b) a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomers,

wherein the interfacial polymerization takes place in the presence of an amine salt.

15. A method of desalting water comprising:

applying, to a liquid, a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of:

(a) a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, or

(b) a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomers,

wherein the interfacial polymerization takes place in the presence of an amine salt.

16. A method of producing ultra-pure water comprising:

applying, to a liquid, a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of:

(a) a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, or

(b) a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomers,

wherein the interfacial polymerization takes place in the presence of an amine salt.

17. A method of desalting an aqueous solution comprising:

applying, to a liquid, a composite semipermeable membrane comprising a porous base material having formed thereon a semipermeable thin film formed by crosslinking interfacial polymerization of:

(a) a 2,6-diaminotoluene monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomer, or

(b) a mixture of a 2,6-diaminotoluene monomer and another amino compound monomer with a polyfunctional crosslinking agent having at least two functional groups capable of reacting with amino groups of the monomers,

wherein the interfacial polymerization takes place in the presence of an amine salt.

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DOCUMENT-IDENTIFIER: US RE36914 E

**** See image for Certificate of Correction ****TITLE: Dialysate filter including an asymmetric microporous, hollow fiber membrane incorporating a polyimideAbstract Text (1):

A dialysate filter including asymmetric, microporous, hollow fiber membranes incorporating a polyimide. The dialysate filter connects to the dialysis machine immediately before the dialyzer ensuring complete filtration of the dialysate, easy visual inspection, quick installation and removal, and easy disinfection of the filter.

Brief Summary Text (3):

This invention relates generally to a dialysate filter which is easy to install, durable and cost effective. In particular, the invention relates to a dialysate filter which removes bacteria and endotoxins from a dialysate stream before it enters an artificial kidney. Still further, the dialysate filter of the present invention includes improved asymmetrical, microporous, hollow fibers incorporating a polyimide.

Brief Summary Text (9):

The prior art has promoted a number of alternatives for removing endotoxins from dialysate. These alternatives include an artificial kidney, ultrafiltration apparatuses, hemo-filters and in-line water filters. These alternatives suffer from a number of disadvantages including difficulty in use, expense and safety risks.

Brief Summary Text (15):

The filter of the present invention includes a housing having an inlet dialysate port, an outlet dialysate port and an access port which function to allow air to be removed and a disinfecting agent to be introduced. The housing contains an asymmetric microporous hollow fiber membrane which incorporates a polyimide.

Drawing Description Text (10):

FIG. 7 illustrates the pressure drop versus conductivity for a polyimide filter.

Drawing Description Text (11):

FIG. 8 illustrates a comparison of pressure drop in water for a variety of filters.

Drawing Description Text (12):

FIG. 9 illustrates a comparison of pressure drop in dialysate between polyimide containing filters and polysulfone containing filters.

Detailed Description Text (2):

The present invention is directed to a filter membrane, a dialysate filter and a dialysis apparatus. Although the membrane and dialysate filter are discussed in connection with their use in dialysis, the skilled artisan would clearly recognize the applicability of the membrane and filter to other technology areas. These areas include water filtration, as a polishing filter for pharmaceutical production, as a plasma filtering device, as a chemofilter, as a hemoconcentrator, and the like.

Detailed Description Text (3):

Dialysate as used herein refers to the final solution, blended within the dialysis apparatus, from sodium bicarbonate concentrate, acid electrolyte concentrate, and ultra-pure water, which flows in a single pass through the dialysate filter and artificial kidney and then finally to drain.

Detailed Description Text (4):

An in-line filter, installed in the dialysate line of a dialysis apparatus, will block passage of most endotoxins contained within reverse osmosis water or the final dialysate stream of the dialysis apparatus. The filter will prevent high loads of endotoxin from reaching the dialysate compartment of the artificial kidney during dialysis and causing Pyrogen Reactions in dialysis patients.

Detailed Description Text (10):

The fiber membrane contained within the housing may be made of any highly permeable filter medium, for example, polymeric fibrous membranes. These membranes can be formed of polysulfone, polycarbonate, polyimide and the like. The filter is preferably made up of asymmetric microporous hollow fiber membranes.

Detailed Description Text (11):

In one preferred embodiment of the present invention, the filter contains asymmetrical microporous, hollow fiber membranes that include a polyimide polymer that is highly polar. We define microporous to mean membranes having a pore size ranging from about 0.005-0.2. μ m. We also define "flux" or "water permeability" to mean a measure of the volume of water passed by the hollow fiber membrane under pressure for a given time and area. "Rewetting" and similar words such as rewettable, rewettability, etc., as used herein, is a description of the ability of a membrane to maintain a particular level of flux or water permeability after either cycles of wetting and drying the membrane or after steam or chemical sterilization. "Asymmetric" means that the pore size of the fiber varies from smaller to larger from the inner barrier layer to the outer sponge-like layer, respectively. "Uniformly porous" and "sponge-like" means that the porosity of the hollow fiber membrane is homogeneous throughout. In addition, "solvents with respect to the polymer" are typically aprotic solvents while "non-solvents with respect to the polymer" are typically protic solvents. "Anti-solvent" is a nonsolvent with respect to the polymer and is used herein when referring to additional nonsolvents that are added to the polymeric solution. "Nonsolvents," on the other hand, are also nonsolvents with respect to the polymer, but is used herein when referring to nonsolvents added to the precipitating solution.

Detailed Description Text (12):

The highly polar polymer in accordance with the present invention is preferably an aromatic polyimide that when precipitated as a membrane is immediately wettable without the use of polymer additives or surfactants. The preferred polyimide in accordance with the present invention is disclosed in U.S. Pat. No. 3,708,458 to Alberino which is incorporated herein by reference, in its entirety. The polyimide is prepared from benzophenone3,3',4,4'-tetracarboxylic acid dianhydride and a mixture of 4,4' -methylenabis(phenylisocyanate) and toluene diisocyanate (2,4- or 2,6-isomer) or mixtures thereof. The polyimide includes the recurring group: ##STR1## wherein 10% to 90% of the R groups are ##STR2## and the remaining R groups include either ##STR3##

Detailed Description Text (13):

The aromatic iso- and diisocyanates may be substituted by their amine analogs. The CAS Registry No. of the preferred polyimide is 58698-66-1. The polyimide is available from Lenzing Corp. (Austria) under the P84 and/or HP P84 (high purity) marks. In an alternative embodiment, a polymer based on the phenylindane diamine; 5 (6)-amino-1-(4'-aminophenyl)-1,3-trimethylindane with a CAS Registry No. of 62929-02-6 may be used. The alternative embodiment polymer is available from Ciba-Geigy

Corporation (Hawthorne, N.Y.) under the Matrimid 5218 mark.

Detailed Description Text (16):

The polyimide polymers useful in accordance with the present invention preferably have a molecular weight of about 30,000 to 125,000 daltons. More preferably, the molecular weight is about 35,000 to 115,000 daltons and most preferably, the molecular weight is about 40,000 to 105,000 daltons.

Detailed Description Text (17):

As stated previously, no additional additives, such as polyvinylpyrrolidone, polyethylene glycol, glycerine, cellulose or starch derivatives or amphoteric, zwitterionic, nonionic, anionic, or cationic surfactants, are needed to produce a hollow fiber membrane that wets immediately upon contact with dialysate, blood, water and other aqueous solutions and maintains the rewettability for at least 6-7 sterilizations by steam or chemicals. Because no additional polymers are needed to make the resultant fiber wettable, the choice of solvents, including nonsolvent combinations at specific ratios, for use as the precipitating solution is critical in influencing the hydrophilicity, structure and porosity of the fiber. In addition, the elimination of additives in the polymeric dope solution decreases and virtually eliminates all but trace amounts of solids and/or oxidizable material that is leachable from the resultant fiber. Further, the structural integrity of the resultant hollow fiber membrane is more stable after the removal of the solvent and/or antisolvents and nonsolvents.

Detailed Description Text (18):

Initially, the polyimide polymer is dissolved in a solvent including solvent/antisolvent combinations. Preferably, this solvent is also miscible with water. A representative, non-limiting list of solvents useful in the invention includes dimethylformamide (DMF), dimethylsulfoxide (DMSO), dimethylacetamide (DMA), n-methylpyrrolidone, and mixtures thereof. Preferably, the solvent is DMF, an aprotic solvent. Depending on the desired properties of the hollow fiber, a small amount of an antisolvent may be added in small quantities to the primary solvent that is used. The addition of an antisolvent in the polymer forming solution will enhance the desired precipitate characteristics of the polymer during fiber formation. For example, adding acetic acid in the amount of 4-7 wt. % ensures that the fiber has a uniform sponge-like structure, free of voids, large vacuous spaces extending from the inner membrane wall to the outer membrane wall that can permit the passage of large molecular weight molecules if the void pierces the inner and/or outer membrane wall. Alternatively, additional amounts of solids may be added to the polymer solution up to 25.0 wt. % to solve this problem. The homogeneous, sponge-like structure may also be achieved in accordance with the process and formulations described herein.

Detailed Description Text (21):

Preferably, about 15-25 wt-%, more preferably, about 16-20 wt-%, and most preferably, about 17-19 wt-% of the fiber forming polyimide polymer is dissolved in the dimethylformamide solvent. When less than 15 wt-% of the polyimide polymer is used, the fibers formed may not be strong enough to withstand the stresses involved in the high speed process in the preferred method of manufacturing the fiber membrane used in the present invention. Further, the fibers lack integrity due to the weakness from the voids in the fiber walls.

Detailed Description Text (22):

Higher polyimide solids may be employed in organic solvent systems if spinnerette housings, feed lines, polymer solution tanks are heated. Upon heating, the viscosity of the polymer solution is lowered, allowing otherwise unusable polymer solution formulations to be spun. Depending upon the composition of the precipitating solution the skilled practitioner chooses, heating and/or cooking the system may influence the morphology and performance characteristics of the resultant fiber membrane.

Detailed Description Text (25):

For example, as the weight percent of the solvent with respect to the polymer increases, fiber formation is impaired and is characterized by a "glassy" weaker structure and it becomes increasingly difficult to "pull" the fiber. Conversely, as the weight percent of the solvent with respect to the polymer decreases and the weight percent of water and/or other non-solvents with respect to the polymer increases, voids are seen in the fiber structure which may allow high molecular weight molecules to pass through the fiber if they pierce the outer membrane wall. This may best be seen in FIG. 6 which illustrates a fiber cross-section magnified 130.times. with voids 221 that resulted from using a precipitating solution with an increased weight percent of non-solvent with respect to the polymer. In addition, as the weight percent of water and/or other non-solvents with respect to the polymer increases, a low pore density on the outer fiber wall and a tighter closed inner wall with a low flux is seen. It will therefore be appreciated by those skilled in the art that the selection of the composition of the precipitating solution is crucial.

Detailed Description Text (26):

The composition of the precipitating solution effective to produce a hollow fiber membrane for use in hemodialysis, as well as, water filters, autologous blood filters, and plasma filters is illustrated below in Table I.

Detailed Description Text (28):

The water which may be used in the precipitating solution may be tap water, deionized water or water which is a product of reverse osmosis. Preferably the water has first been treated by reverse osmosis.

Detailed Description Text (30):

Additional combinations of solvents and non-solvents, which may or may not contain salts, may be used so long as they are miscible with dimethylformamide, dimethylsulfoxide, dimethylacetamide, n-methylpyrrolidone and mixtures thereof. A representative, non-limiting list of non-solvents (with respect to the polymer) that may be used in the precipitating solution are acetic acid, isopropanol, water, glycerol, acetic anhydride, and ethanol.

Detailed Description Text (31):

The proportions of the water, and other non-solvents (e.g. alcohol) which may make up the precipitating solution influence the morphology, clearance, permeability, and selectivity characteristics of the hollow fiber membrane. In particular, the total absence of a solvent with respect to the polymer in the precipitating solution may result in a small number of pores in the fiber wall as well as lower flux. Further, water is clearly an important ingredient in the precipitating solution used in this membrane formation process.

Detailed Description Text (32):

Because the addition of water affects the performance characteristics of the resultant fiber membrane it is generally preferred that the proportion of water in the precipitating solution be about 1-35 wt. %, to ensure proper fiber performance characteristics. Less than about 10 wt. % of water may result in the polymeric solution precipitating too slowly forming a fiber with increased pore size. This is desirable to form a fiber for use in water filters but would not, for example, form a fiber suitable for use as a dialyzer fiber. Conversely, a concentration of water greater than about 35 wt. % results in a fiber with lower pore density on the outside and a tighter closed inner wall with a general decrease in flux. However, when the proportion of water falls within 1-35 wt. %, we see enhanced uniformity in the desirable sponge-like structure and the hollow fiber membrane is characterized by the complete absence of voids. This uniformity results in more overall uniform flux with respect to all types of filters and tighter controls with respect to molecular weight cutoffs in dialyzer applications.

Detailed Description Text (34):

Initially, the highly polar polymer is diluted in DMF. Depending on the desired properties and characteristics of the hollow fiber, a small amount of a non-solvent (with respect to the polymer) (also called anti-solvents) other than water may be added instead of using pure DMF solvent. This may enhance the precipitation of the polymer in the fiber formation. For example, the addition of 4-7 wt. % glacial acetic acid to the polymer/DMF solution enhances the uniform sponge-like structure of the resultant fiber and the fiber is further characterized by the complete absence of voids.

Detailed Description Text (43):

The fiber is submerged in a tank comprising water and 0-10 wt. % other materials. Again, the water may be tap, or any purified water including deionized water, or the product of a reverse osmosis process. The temperature of the quenching bath is preferably between about 0.degree. C. to 100.degree. C., more preferably, about 15.degree. C. to 45.degree. C., and most preferably, about 35.degree. C. The water temperature directly affects the performance of the fiber. Lower temperatures can reduce the flux of the resulting fiber. Increasing the quenching bath temperature can increase the flux of the fiber.

Detailed Description Text (44):

The fiber is preferably immersed in the quenching bath for a period of about 0.1 to 10 seconds, preferably about 0.1 to 5 seconds, and most preferably, about 1 second. This residence time permits the full precipitation of the polyimide polymer to form the microporous hollow fiber.

Detailed Description Text (45):

After the quenching bath, the fiber may be further rinsed to remove any remaining solvents. This rinsing may be accomplished in a water bath arrangement. Preferably, the additional rinse is achieved in a water bath having a water temperature of about 0.degree. C.-100.degree. C., more preferably, about 15.degree. C.-45.degree. C., and most preferably, about 35.degree. C. The fiber is then wound on a take-up reel. The take-up reel is preferably rotating at a speed such that the fiber is being wound at about 90-175% of the rate at which it is being formed at the spinnerette or, in other words, at approximately about 150-250 ft/min (about 45-77 m/min). More preferably, the fiber is being wound at a rate substantially equal to that at which it is being produced. In other words, the fiber is taken up with enough speed (i) to create a fiber of the desired size and (ii) to apply sufficient tension to the fiber such that it will remain taut in the take-up guide unaffected by ambient air currents, i.e. there is no "draft."

Detailed Description Text (46):

The hollow fibers may then be dried by any method appropriate to general manufacturing procedures including but not limited to air, heat, vacuum, or any combination thereof. The hollow fibers may be further processed to form useful articles including hemodialyzer cartridges, hemofilters, blood filters, water filters, etc., having improved performance levels.

Detailed Description Text (47):

In the present invention, polyimide fibers are preferred over polysulfone because they have a lower pressure drop across the filter, i.e., they are more permeable.

Detailed Description Text (59):

Examples 1-7 characterize and describe how to prepare the polyimide fibers according to one preferred embodiment of the present invention. Examples 8-29 describe the preparation, testing, cleaning and use of the filters of the present invention.

Detailed Description Text (61):

A polymeric dope solution was formed by dissolving 17.5 wt. % of P84 in dimethylformamide. The material was filtered and then pumped to a tube-in-orifice spinnerette at a rate of 4.50 mL/min and at a temperature of 24.degree. C. Simultaneously, a precipitating solution consisting of 80 wt. % dimethylformamide and 20 wt. % reverse osmosis deionized water was mixed, filtered and delivered to the spinnerette at a temperature of 24.degree. C. and a rate of 2.75 mL/min.

Detailed Description Text (62):

The polymeric dope solution was delivered through the outer, annular orifice of the spinnerette, which orifice had an outside dimension of about 0.022 to 0.025 inches (about 560 .mu.m) and an inside dimension of about 0.010 inches (about 254 .mu.m). The precipitating solution was delivered through a tube-in-orifice within the annular orifice, which tube-in-orifice had an inside diameter of about 0.005 inches (about 127 .mu.m). The spinnerette head was maintained at 24.degree. C. The spinnerette discharged the polymeric solution and precipitating solution downward into ambient atmosphere for a distance of about 1.5 meters into a quenching bath maintained at 32.degree. C. Formed fiber material was wound on a take-up reel at a rate of 70 m/min. The fiber was then removed from the take-up wheel, cut, bundled, soaked in a water bath at 32.degree. C. for 10 hours, dried and tested.

Detailed Description Text (66):

The method for preparing fiber as in Example 1 was repeated using a precipitating solution of 81 wt. % DMF and 19 wt. % deionized water.

Detailed Description Text (70):

The method employed in Example 1 was repeated using 17.0 wt. % of the P84 polyimide polymer and 83 wt. % DMF. The precipitating solution comprised 81 wt. % DMF and 19.0 wt. % deionized water. Sieving-coefficients were similar to the Test Data obtained for Examples 1 and 2 above.

Detailed Description Text (72):

Fibers for use in a water filter were manufactured in the following manner. A polymeric dope solution was formed by dissolving 19.0 wt. % of Matrimid 5218 in 81.0 wt. % DMF. The material was filtered and then pumped to a tube-in-orifice spinnerette at a rate of 2.9 mL/min at a temperature of 23.degree. C. Simultaneously, a precipitating solution consisting of 85.5 wt. % DMF and 14.5 wt. % water was mixed, filtered and delivered to the spinnerette at a temperature of 23.degree. C. and a rate of 3.0 mL/min.

Detailed Description Text (73):

The polymeric dope solution was delivered through the outer, annular orifice of the spinnerette having an outside diameter of 940 .mu.m and an inside diameter of 254 .mu.m. The precipitating solution was delivered through a tube-in-orifice within the annular orifice having an inside diameter of about 127 .mu.m. The spinnerette head was maintained at about 23.degree. C. The spinnerette discharged the column of polymeric/solution and precipitating solution downward for a distance of about 0.81 m into a quenching water bath maintained at a temperature of 35.degree. C. The fiber was wound on a take-up reel at a rate of about 45 m/min. Cut bundles were soaked in a 46.degree. C. water bath for 16 hours. Fiber bundles were dried and tested. Based on a 0.05 m.sup.2 test mat, at 5 psi, water permeability was calculated to be 500 mL/(hr.times.m.sup.2 .times.mmmHg).

Detailed Description Text (75):

Fibers for use in a plasma filter were manufactured in the following manner. The method for preparing fiber as in Example 4 was repeated using a polymeric dope solution consisting of 16.75% P84 polymer and 83.25 wt. % DMF. The precipitating solution included 85.5 wt. % DMF and 14.5 wt. % deionized water. Fibers had a sieving coefficient of 0.65 using a 0.1% solution of fluorescein isothiocyanate dextran (Sigma), a molecular weight marker of approximately 500,000 Daltons. Water permeability was in excess of 900 (mL/hr/mmmHg/m.sup.2).

Detailed Description Text (77):

Fibers for use in a water filter were manufactured in the following manner. A polymeric dope solution was formed by dissolving 16.75 wt. % P84 polymer in 83.25 wt. % DMF. The material was filtered and then pumped to a tube-in-orifice spinnerette at a rate of 4.5 mL/min at a temperature of 23.degree. C. Simultaneously, a precipitating solution consisting of 85.5 wt. % DMF and 14.5 wt. % water was mixed, filtered and delivered to the spinnerette at a temperature of 23.degree. C. and a rate of 3.0 mL/min.

Detailed Description Text (78):

Fibers were further processed in accordance with the method of Example 4. .[.Fibers were further processed in accordance with the method of Example 4..]. A water filter (1.5 m.sup.2 of fiber) containing the fibers manufactured using the above formulation was tested for water permeability. At 8.6 psi, filters had a water permeability of 1020 ml/(hr.times.m.sup.2 .times.mmmHg). At 10.0 psi, filters had a water permeability of 1320 ml/(hr.times.m.sup.2 .times.mmmHg).

Detailed Description Text (80):

Fibers for use in water filters were prepared in the following manner. A polymeric dope solution was formed by dissolving 15.2 wt. % P84 polyimide polymer in 79.80 wt. % DMF and 5.0 wt. % glacial acetic acid. The material was filtered and pumped to a tube-in-orifice spinnerette at a rate of 4.1 mL/min. A precipitating solution comprised of 50 wt. % DMF and 50 wt. % glacial acetic acid was mixed, filtered and delivered to the spinnerette at a rate of 4.5 mL/min.

Detailed Description Text (81):

The polymeric dope solution was delivered through the outer, annular orifice of the spinnerette having an outside dimension of about 0.029 inches (737 .mu.m) and an inside dimension of about 0.01 inches (about 254 .mu.m). The precipitating solution was delivered through a tube-in-orifice within the annular orifice having an inside diameter of about 0.005 inches (about 127 .mu.m). Precipitated fiber was quenched in a reverse osmosis water bath and taken up at a rate of 49 m/min.

Detailed Description Text (82):Water PermeabilityDetailed Description Text (83):

All fibers produced in the Examples above were evaluated for water permeability (flux) in the following manner. Water was passed through the lumens of potted test fibers with the filtering unit in a horizontal position. The ultrafiltrate port on the inlet side of the unit was plugged. Pressure monitors were placed at all inlet and outlet ports. With flow through the unit, back pressure was applied to the fiber outlet side of the unit to increase ultrafiltrate flow across the fibers. Three data points were taken at 10%, 50%, and 80-100% ultrafiltrate flow and transmembrane pressure (TMP) was calculated. Ultrafiltrate flow was plotted against TMP and the slope of this curve was used to determine flux or water permeability. As noted above, all of the above fibers for use as water filters, hemofilters and dialyzers had water permeabilities in excess of 75.times.10.sup.-5 ml/(min.times.cm.sup.2 .times.mmmHg).

Detailed Description Text (85):

A dialysate filter was prepared by spinning hollow fibers incorporating a polyimide. The dried fiber bundles were inserted into the molded polycarbonate case. Special potting caps were placed at the ends of the molded polycarbonate case. The ends of each fiber were cut to size. Urethane potting material was then centrifugally placed in the case to seal the fibers to the case. Excess potting material was cut away to expose the ends of the fibers and provide a fluid flow path in the completed dialysate filter. Once the ends of the filter were potted and cut, polycarbonate headers fitted with an O-ring were placed on the ends of the

case. A cap was placed over the access port. One end of the filter was resealed with urethane so that the dialysate only had one outlet from the unit. The unit's maximum levels of residuals were 25 ppm for ethylene oxide, 25 ppm for ethylene chlorohydrin and 250 ppm for ethylene glycol. The unit was packaged in a sealed Tyvek pouch. The filter had the following properties:

Detailed Description Text (89):

The electrolyte composition was analyzed at the filter inlet and outlet for a dialysate filter as described in Example 8. The dialysate filter was installed on a Travenol 450 SPS dialysis machine between the dialyzer-inlet and dialyzer-outlet lines. A throughput flow at 600 ml/min was initiated and concentrate uptake lines were connected to sterile, non-pyrogenic liquid bicarbonate and acid electrolyte concentrate containers. Concentrates were diluted internally by the Travenol 450 SPS using reverse osmosis water meeting the AAMI standards for water used to make dialysate.

Detailed Description Text (92):

Changes to solution in terms of conductivity/resistivity and extrapolate to parts per million (or parts per billion) amounts of material contributed by a filter as described in Example 8 into the filtrate, using ultrapure de-ionized water as a filter throughput solution.

Detailed Description Text (93):

One-at-a-time, two filters were installed in a pressurized DI water line with a resistivity monitoring cell before and after the filter. The control valve was slowly opened and 1,000 ml/min flow of DI water through the filter was obtained and maintained. The inlet and outlet resistivity cell values were read and recorded at least every five minutes for a 30 minute period. The values were averaged for the two filters to arrive at a single inlet DI water resistivity value and a single outlet water resistivity value. The resistivity values were converted to ppm (or ppb) and the inlet value was subtracted from the outlet value to determine approximate ionic content added to DI water from the filter. Resistivity values were averaged over the 30 minute period and between the two filters used. Filtrate conductivity=0.169 $\mu\text{S}/\text{cm}$ (or 0.37 ppm). This was an increase from the inlet DI water conductivity of 0.066 $\mu\text{S}/\text{cm}$ (or 0.145 ppm). Therefore, approximately 0.224 ppm (or 224 ppb) of unknown ionic shedding or release of dissolved solids can be attributed to the filter.

Detailed Description Text (95):

The filter inlet and outlet pressures (mmHg) were observed in water and final bicarbonate-based dialysate for a filter as described in Example 8. The transmembrane pressure drop was also examined as it related to the filter's effect upon the dialysate stream pressures and flow rates. A filter as described in Example 8 was installed on a Travenol 450 SPS dialysis machine between dialyzer-in and dialyzer-out dialysate lines. The sample port Tees, with 3-way stopcocks attached were installed in the upstream and downstream lines. Tubing was attached between the stopcocks and the Digi-dyne.sup.- pressure monitor transducers (one for filter inlet pressure and one for filter outlet pressure). The stopcocks were opened to the monitoring tubing and turned on the pressure monitors.

Detailed Description Text (96):

RO water throughput at a flow at 600 ml/min was initiated. Transmembrane pressure drop was observed across the filter. The nature of the filter and whether or not the filter functioned as a flow restrictor was detected by measuring flow in the drain line with a graduated cylinder over a one minute period.

Detailed Description Text (97):

With the dialysis machine still running at 600 ml/min and monitor lines still open, dialysate throughput flow was initiated by connecting concentrate uptake lines to sterile, non-pyrogenic liquid bicarbonate and acid electrolyte concentrate

containers. The acid concentrate used .[.were.]. .Iadd.was .Iaddend.Renal Systems SB-1075 and the bicarbonate concentrate used was BC-1-L. Concentrates were diluted internally by the Travenol 450 SPS using RO water meeting the AAMI standards for water used to make dialysate.

Detailed Description Text (100):

As shown in the FIG. 7, the filter displayed a one-time variance in pressure drop that is corrected the first time each filter contacts final dialysate solution. The initial transmembrane pressure drop at 600 ml/min in RO water is about 260-340 mmHg initially. Within a few minutes, once the membrane is thoroughly "wetted", the TMP drop falls to about 200 mmHg and is stable. When contact with ion-rich dialysate occurs, the figure shows how we cycled back and forth between RO water and dialysate three times to illustrate the permanence of the change in pressure drop once dialysate contact occurs. At no time did the filter function as an actual in-line flow restrictor.

Detailed Description Text (102):

Several different hollow fiber membrane devices were installed, one-at-a-time on a dialysis machine, under identical operating conditions, and the differences in transmembrane pressure drop in water and dialysate were determined for each device..

Detailed Description Text (104):

RO water throughput flow at 600 ml/min was initiated and maintained for at least five minutes to allow conditions to stabilize. The flow rate was verified by measuring flow in the drain line with a graduated cylinder over a one minute period. The inlet and outlet pressures were recorded and the transmembrane pressure drop across the filter was calculated.

Detailed Description Text (105):

With the dialysis machine still running at 600 ml/min and monitor lines still open, dialysate throughput flow was initiated by connecting concentrate uptake lines to sterile, non-pyrogenic liquid bicarbonate and acid electrolyte concentrate containers. The acid concentrate used was Renal Systems.RTM. SB-1075 and the bicarbonate concentrate used was BC-1-L. Concentrates were diluted internally by the Travenol 450 SPS using RO water exceeding the AAMI standards for water used to make dialysate.

Detailed Description Text (107):

Steps one through five were repeated for a dialyzer using the same membrane as the RenaGuard.TM. Dialysate Filter (polyimide), a Minntech Primus.RTM.1350 dialyzer (polysulfone), and a Fresenius F60 dialyzer (polysulfone).

Detailed Description Text (108):

Both the RenaGuard.TM. Dialysate Filter and a dialyzer made with polyimide fibers had lower pressure drops than both of the Minntech and Fresenius polysulfone units. The results are set forth in FIG. 8. Polyimide fiber units had a lower pressure drop in dialysate than in RO water. The polysulfone units, on the other hand, had a higher pressure drop in dialysate.

Detailed Description Text (131):

Prior to installation of the dialysate filter, the Travenol machine had been treated with 250 ml of Renalin.RTM. Concentrate diluted within the dialysis machine 1:8 in RO water and allowed to dwell within the machine for 30 minutes. After this time, the machine was allowed to rinse until peracetic acid test strips indicated <1 ppm in the drain line. Peroxide levels from the Renalin.RTM. were also verified <1 ppm in filter outlet samples as detected by using a Spectrophotometer.

Detailed Description Text (136):

Two dialysate filters as described in Example 8 were installed on separate Travenol

450 SPS dialysis machines and left attached between the dialysate-inflow and dialysate-outflow lines. Each filter was exposed to over 600 liters of throughput, 500 liters of which were properly proportioned final bicarbonate-based dialysate, the remainder being RO water. The throughput flow rate was 600 ml/min.

Detailed Description Text (138):

Prior to initiating the bacterial challenge, each Travenol machine had been treated with 250 ml of Renalin.RTM. Concentrate diluted within the dialysis machine 1:8 in RO water and allowed to dwell within the machine for 30 minutes. After this time, the machine was allowed to rinse until peracetic acid test strips indicated <1 ppm in the drain line. Peroxide levels from the Renalin.RTM. were also verified <1 ppm in filter outlet samples as detected by using a Spectrophotometer.

Detailed Description Text (149):

a. One filter was challenged in both DI water and dialysate. This filter received two back-to-back endotoxin challenge tests.

Detailed Description Text (153):

e. One filter had been "pre-treated" by installing it as a pre-filter for the Renatron.RTM. artificial kidney reprocessing machine. It was exposed to RO water throughput conditions of up to 6.2 liters/min (6,200 ml/min) during ten (10) Renatron.RTM. processing cycles.

Detailed Description Text (154):

f. Again, one filter was installed on a dialysis machine as described above and exposed to throughput flowrates of 600 ml/min. This dialysis machine picked up the endotoxin contamination from the water inlet to the machine, instead of from contaminated dialysate. Frozen, dried Pseudomonas aeruginosa LPS, was reconstituted per the manufacturer's instructions and added to the water supply for the dialysis machine (a 50-liter polyethylene container). To provide adequate pressure for dialysis machine operation, a roller pump "pushed" the contaminated water stream into the dialysis machine.

Detailed Description Text (157):

Six (6) ETO'd filters as described in Example 8 were challenged with endotoxin in RO water at flowrates of 2,000 ml/min and verify endotoxin retention.

Detailed Description Text (158):

Six filters were individually removed from the Tyvek bag used during the ETO sterilization process and installed in the test setup for one-at-a-time evaluation. RO water was used as the challenge solution due to the difficulty presented in producing large volumes of clean dialysate to support the 2 liters per minute flow rate used in this test.

Detailed Description Text (160):

a. RO water was used instead of final bicarbonate-based dialysate.

Detailed Description Text (167):

Filters received endotoxin challenges varying in potency from .gtoreq.15 EU/ml up to 48 EU/ml; of either Escherichia coli or Pseudomonas aeruginosa endotoxin at RO water throughput flowrates of 2,000 ml/min. Each filter retained all endotoxin contained within the challenge solution. No detectable endotoxin is reported as <0.06 EU/ml, the limit of sensitivity for the Lysate used in the assay. (See Table below for details.)

Detailed Description Text (173):

We had observed, that at a point between 10,000 liters and 20,000 liters total dialysate throughput, the filters began "shedding" of LAL-reactive material into the filtrate. Concurrently, the pressure drop across the filters had doubled. That is, they showed less permeability to throughput fluids, and no longer served to

depyrogenate the fluids flowing through them. When pyrogen-free RO water was input to the filter the fluid became LAL-reactive.

Detailed Description Text (179):

c. Running RO water through the filter at 600 ml/min during a Renalin.RTM. Concentrate chemical treatment on the dialysis machine.

Detailed Description Text (182):

3) Turning on the dialysis machine and "rinsing" the filter with RO water until <1 ppm of Renalin.RTM. was detected. This took about 20 minutes to accomplish.

Detailed Description Text (185):

This endotoxin test setup consisted of a roller pump, inlet and outlet tubing and Hansen-style connectors, pre- and post-filter sampling ports, and 2 1/2 gallon polyethylene containers for holding the challenge solutions. All tests used DI water throughput at a flowrate=500 ml/min.

Detailed Description Text (188):

Frozen, dried lipopolysaccharide (LPS, endotoxin), isolated from Escherichia coli by phenol extraction, was reconstituted per the manufacturer's instructions and added to a 2 1/2 gallon container of pyrogen-free RO water immediately prior to initiating the test. The 2 1/2 gallon container was vigorously shaken and then placed on a magnetic stirring platform, with a stirring bar in the bottom, to help keep the solution mixed.

Detailed Description Text (191):

Filters then received follow-up endotoxin challenges varying in potency from .gtoreq.7.7 EU/ml up to 30.7 EU/ml of Escherichia coli endotoxin at RO water throughput flowrates of 500 ml/min. Each filter retained all endotoxin contained within the challenge solution. No detectable endotoxin was reported as <0.06 EU/ml, the limit of sensitivity for the Lysate used in the assay. (See Table below for details.)

Detailed Description Text (198):

This 200 ml of effluent solution was aseptically filtered through a 0.2 .mu.m membrane filter. It was then rinsed with 90 ml of 1% peptone/1% sodium thiosulfate solution to neutralize the Renalin.RTM., followed by 100 ml sterile DI water.

Detailed Description Text (199):

The effectiveness of this 90 ml 1% peptone/1% sodium thiosulfate neutralizing "rinse" procedure was verified. By contaminating the 100 ml sterile DI water used as a rinse (above) with Bacillus subtilis spores, plating the 0.2 .mu.m filter on TSA, and incubating at 37.degree. C. for 24 hours, we were able to show bacterial growth. This indicates effective neutralization of the Renalin.RTM. used in the test.

Detailed Description Text (203):

First a test was conducted to demonstrate the recoverability of Bacillus subtilis spores impacted upon the dialysate filter membrane. An ETO-sterilized dialysate filter was installed on a Travenol 450 SPS dialysis machine between the dialyzer-in and dialyzer-out dialysate lines. RO water flow was initiated through the filter at 600 ml/min. One ml of about 10.sup.10 Bacillus subtilis spores [.were.]. .Iadd.was .Iaddend.injected into the filter's inlet stream. The dialysis machine was allowed to run for about 5 more minutes. Then, by reversing the filter's installation configuration we were able to "blow off" the spores embedded or impacted upon the membrane. Recovery was estimated to be nearly 100%.

Detailed Description Text (204):

Then, each of the four filters to be used in the sporicidal Renalin.RTM. treatment test was installed one-at-a-time on a Travenol 450 SPS dialysis machine between the

dialyzer-in and dialyzer-out dialysate lines. RO water flow was initiated through each filter at 600 ml/min. One ml containing 10.sup.10 spores was injected into the filter's inlet stream and the machine was allowed to run for another 5 minutes to drive the spores against the membrane. The machine was turned off and 10 ml of Renalin.RTM. Concentrate was injected into the Luer port on each filter. The filters remained installed on the dialysis machine for a minimum of six hours.

Detailed Description Text (207):

Filtrate was then passed through a sterile 0.2 .mu.m Nalgene analytical filter, followed by at least 90 ml of the "neutralizing rinse" used in the previous test. The neutralizing rinse was then followed by at least 100 ml of sterile DI water.

Detailed Description Text (216):

This 200 ml of effluent solution was aseptically filtered through a 0.2 .mu.m membrane filter. It was then rinsed with 90 ml of 1% peptone/1% sodium thiosulfate solution to neutralize the Renalin.RTM., followed by 100 ml sterile DI water.

Detailed Description Text (222):

We first conducted a test to demonstrate the recoverability of Bacillus subtilis spores as it impacted upon the dialysate filter membrane. An ETO'd dialysate filter was installed on a Travenol 450 SPS dialysis machine between the dialyzer-in and dialyzer-out dialysate lines. RO water flow was initiated through the filter at 600 ml/min. One ml of about 10.sup.10 cfu/ml Bacillus subtilis spores .[.were.]. .Iadd.was .Iaddend.injected into the filter's inlet stream. The dialysis machine was allowed to run for about 5 more minutes. Then, by reversing the filter's installation configuration we were able to "blow off" the spores embedded or impacted upon the membrane. Recovery was estimated to be nearly 100%.

Detailed Description Text (223):

Then, each of the four filters to be used in the sporicidal Renalin.RTM. treatment test was installed one-at-a-time on a Travenol 450 SPS dialysis machine between the dialyzer-in and dialyzer-out dialysate lines. RO water flow was initiated through each filter at 600 ml/min. One ml of spore suspension was injected into the filter's inlet stream and the machine was allowed to run for another 5 minutes to drive the spores against the membrane. The machine was turned off and Renalin.RTM. Concentrate was injected into the Luer port on each filter. The filters remained installed on the dialysis machine for a minimum of six hours with Renalin.RTM. indwelling.

Detailed Description Text (225):

Filtrate was then passed through a sterile 0.2 .mu.m Nalgene analytical filter, followed by at least 90 ml of the "neutralizing rinse" used in the previous test. The neutralizing rinse was then followed by at least 100 ml of sterile DI water.

Detailed Description Text (232):

DI water throughput was initiated at 500 ml/min and continued for the duration of the test. Endotoxin derived from Escherichia coli was introduced into the filter's inlet stream at 15.4 EU/ml. Filtrate

Detailed Description Text (235):

With 500 ml/min DI water throughput, containing a 15.4 EU/ml Escherichia coli endotoxin level, no endotoxin was detected in filter outlet samples at 1, 3, and 5 minutes. No detectable endotoxin was recorded as <0.06 EU/ml, the limit of lysate sensitivity to the presence of endotoxin. The pressure drop across the filter did not change from values recorded prior to heat treatment exposures.

Detailed Description Text (238):

Three filters were exposed to full-strength bleach (5.25% sodium hypochlorite) during 600 ml/min throughput of either RO water, acetate dialysate, or final bicarbonate-based dialysate solutions using a Travenol 450 SPS dialysis machine.

Eight injections of undiluted bleach, 20 cc each, were delivered into the inlet stream of each filter at 15 minute intervals.

Detailed Description Text (239):

Two filters were exposed to approximately 30 days' worth of bleach treatments. Both filters were given six exposures of 1,250 ml each, with a 5 minute rinse between each exposure. The dilution level was 1:8 bleach in RO water. The dilution was accomplished by using a COBE Centry 2Rx dialysis machine. The total bleach contact time was 2 hours and 5 minutes. The filters were installed on the dialysis machine and the bleaching procedure followed right out of the COBE Centry 2Rx Operator's manual. The only exception was that 7,500 ml of bleach was used instead of the 250 ml specified in the procedure.

Detailed Description Text (240):

All five filters were rinsed with DI water on the dialysis machine until 0 ppm of free chlorine was detected using Hach Co. "powder pillows" for chlorine testing and the color comparator wheel provided with the test kit.

Detailed Description Text (242):

DI water throughput was initiated at 500 ml/min and continued for the duration of the test. Endotoxin derived from Escherichia .[.coil.]. .Iadd.coli .Iaddend.was introduced into the filter's inlet stream at .gtoreq.3.8 EU/ml. Filtrate endotoxin levels were measured using the gel-clot method of Limulus Amebocyte Lysate endotoxin assaying.

Detailed Description Text (247):

The third filter, exposed to RO water throughput and eight 20 cc bleach injections, was challenged with .gtoreq.7.7 EU/ml level of Escherichia coli endotoxin. No endotoxin was detected in filter outlet samples at 5, 30, and 60 minutes.

Detailed Description Text (253):

One filter was exposed to approximately 30 days' worth of Actril.RTM. treatments. The filter was given six exposures of 1,250 ml each, with a 5 minute rinse between each exposure. The dilution level was 1:8 Actril.RTM. in RO water. The dilution was accomplished by using a COBE Centry 2Rx dialysis machine. The total Actril.RTM. contact time was 2 hours and 5 minutes. The filter was installed on the dialysis machine and the procedure followed right out of the COBE Centry 2Rx Operator's manual. The only exceptions were that 7,500 ml of chemical was used instead of the 250 ml specified in the procedure, and, Actril.RTM. was used instead of bleach.

Detailed Description Text (254):

The filter was rinsed with DI water on the dialysis machine until <1 ppm of hydrogen peroxide was detected using Renalin.RTM. residual test strips (for hydrogen peroxide, not peracetic acid).

Detailed Description Text (256):

DI water throughput was initiated at 500 ml/min and continued for the duration of the test. Endotoxin derived from Escherichia coli was introduced into the filter's inlet stream at .gtoreq.15.4 EU/ml. Filtrate endotoxin levels were measured using the gel-clot method of Limulus Amebocyte Lysate endotoxin assaying.

Detailed Description Text (258):

With 500 ml/min DI water throughput, containing a 15.4 EU/ml Escherichia coli endotoxin level, no endotoxin was detected in filter outlet samples at 1, 3, and 5 minutes. No detectable endotoxin is recorded as <0.06 EU/ml, the limit of lysate sensitivity to the presence of endotoxin. The pressure drop across the filter did not change from values recorded prior to Actril.RTM. treatment exposures.

Detailed Description Text (261):

Four dialysate filters that had been exposed to conditions which caused them to

release LAL-reactive material into throughput solutions, were treated with a 250 ml 1:8 dilution Renalin.RTM. in RO water solution and reverse flow conditions. Renalin.RTM. residuals were rinsed to <1 ppm hydrogen peroxide. Endotoxin retention capability was re-evaluated after rinsing has been completed.

Detailed Description Text (269):

c. Running RO water through the filter at 600 ml/min during a Renalin.RTM. Concentrate chemical treatment on the dialysis machine.

Detailed Description Text (272):

3) Turning on the dialysis machine and "rinsing" the filter with RO water until <1 ppm of Renalin.RTM. was detected. This took about 20 minutes to accomplish.

Detailed Description Text (275):

This endotoxin test setup consisted of a roller pump, inlet and outlet tubing and Hansen-style connectors, pre- and postfilter sampling ports, and 21/2 gallon polyethylene containers for holding the challenge solutions. All tests used DI water throughput at a flowrate=500 ml/min.

Detailed Description Text (277):

Frozen, dried lipopolysaccharide (LPS, endotoxin), isolated from Escherichia coli by phenol extraction, was reconstituted per the manufacturer's instructions and added to a 21/2 gallon container of pyrogen-free RO water immediately prior to initiating the test. The 21/2 gallon container was vigorously shaken and then placed on a magnetic stirring platform, with a stirring bar in the bottom, to help keep the solution mixed.

Detailed Description Text (279):

Filters subjected to a Renalin.RTM. membrane clearing and disinfecting procedure and receiving follow-up endotoxin challenges varying in potency had the following . [.results, .]. .Iadd.results .Iaddend.with a challenge from .gtoreq.7.7 EU/ml up to 30.7 EU/ml of Escherichia coli endotoxin in RO water at throughput flowrates of 500 ml/min, each filter retained all endotoxin contained within the challenge solution. No detectable endotoxin is reported as <0.06 EU/ml, the limit of sensitivity for the Lysate used in the assay. (See Table below for details.)

Detailed Description Text (281):

A filter as described in Example 8 was exposed to an amount of bleach contact consistent with normal dialysis machine "low-level" disinfection, and, a clearance curve (ppm vs. time) of residual free chlorine levels during 500 ml/min RO water throughput was plotted.

Detailed Description Text (283):

This treatment consisted of an exposure to a 1:8 dilution bleach (0.65% sodium hypochlorite) in water solution while the filter was installed on the dialysis machine.

Detailed Description Text (287):

Free chlorine residuals rinsed from the filter down to 1.6 ppm within 10 minutes at a 470 ml/min RO water throughput flow rate.

Detailed Description Text (289):

A filter as described in Example 8 was exposed to an amount of Actril.RTM. contact consistent with normal dialysis machine "low-level" disinfection, and, a clearance curve (ppm vs. time) of residual hydrogen peroxide levels during 500 ml/min RO water throughput was plotted.

Detailed Description Text (291):

This treatment consisted of an exposure to a 1:8 dilution Actril.RTM. (0.1% hydrogen peroxide) in water solution while the filter was installed on the dialysis

machine.

Detailed Description Text (295):

Hydrogen peroxide residuals rinsed from the filter down to 0.6 ppm within 10 minutes at a 470 ml/min RO water throughput flow rate.

Detailed Description Paragraph Table (1):

TABLE I	More Most Preferred Preferred
Preferred	Solvent with 50-99 wt. % 60-95 wt. % 75-90 wt. % respect to polymer <u>Water</u> 35-1 wt. % 30-5 wt. % 20-10 wt. % Add'l Non-Solvents 15-0 wt. % 10-0 wt. % 5-0 wt. % with respect to polymer

Detailed Description Paragraph Table (9):

TABLE V	Change in Throughput Solution
Resistivity Due to Filters T = 0 Minutes T = 30 Minutes Average Inlet <u>Water</u>	
Filtrate Filtrate Filtrate Resistivity, Resistivity, Resistivity, Resistivity, Filter # Megohm-cm Megohm-cm Megohm-cm Megohm-cm	
	1.00 15.38 7.00 5.70 6.25 2.00 14.55 5.63
5.30 5.48 AVG 15.10 6.32 5.50 5.90	

Detailed Description Paragraph Table (14):

Endotoxin Filtrate Filter Level, Level, EU/ml Treatment # EU/ml 1 MIN 3 MIN 5 MIN conditions 1 6 <0.06 <0.06 <0.06 Also DI water, 48 EU/ml in, <0.06 EU/ml in all outlet samples 2 6 <0.06 <0.06 <0.06 -- 3 12 <0.06 <0.06 <0.06 Pseudomonas LPS sonicated & vortexed prior to use 4 60 <0.06 <0.06 <0.06 Pseudomonas LPS sonicated & vortexed prior to use 5 12 <0.06 <0.06 <0.06 800 ml/min throughput flowrate 6 24 <0.06 <0.06 <0.06 Run on dialysis machine, 600 ml/min LPS introduced into water inlet 7 4.8 <0.06 <0.06 <0.06 Pre-treated with 10 Renatron .RTM. cycles 8 6 <0.06 <0.06 <0.06 Also 1,000 ml/min throughput flowrate, 12 EU/ml inlet, <0.06 EU/ml outlets

Detailed Description Paragraph Table (15):

TABLE X	Syringe Pump Endotoxin Challenge at 2,000 ml/min Endotoxin Endotoxin Challenge Filtrate Filter Level, Level, EU/ml # EU/ml 1 MIN 4 MIN Treatment conditions
	1 48 <0.06 <0.06 E. Coli LPS, contaminated RO <u>water</u> in 2 1/2 gallon jug 2 24 <0.06 <0.06 E. Coli LPS, Syringe pump administration 3 .gtoreq.15 <0.06 <0.06 P. aeruginosa LPS, Syringe pump administration 4 .gtoreq.15 <0.06 <0.06 P. aeruginosa LPS, Syringe pump administration 5 .gtoreq.15 <0.06 <0.06 P. aeruginosa LPS, Syringe pump administration 6 .gtoreq.15 <0.06 <0.06 P. aeruginosa LPS, Syringe pump administration

Detailed Description Paragraph Table (20):

TABLE XIV	Endotoxin Filtrate Challenge
Endotoxin Filter # Pre-treat Conditions Level Level	
	1 Eight injections of 20 cc 7.7 EU/ml <0.06 EU/ml bleach delivered during bicarbonate throughput 2 Eight injections of 20 cc 7.7 EU/ml <0.06 EU/ml bleach delivered during bicarbonate throughput 3 Eight injections of 20 cc 7.7 EU/ml <0.06 EU/ml bleach delivered during RO <u>water</u> throughput 4 30 Days' worth of 3.8 EU/ml <0.06 EU/ml bleach treatments 5 30 Days' worth of 15.4 EU/ml <0.06 EU/ml bleach treatments

Current US Cross Reference Classification (7):

210/500.39

Other Reference Publication (5):

G. B. Harding et al., "Endotoxin and Bacterial Contamination of Dialysis Center

Water and Dialysate: a Cross Sectional Survey", Intl. J. Artificial Organs, vol. 13, No. 1, 1990, pp. 39-43..

CLAIMS:

1. A filter comprising;

a housing defining an interior chamber;

an inlet port connected to said housing;

an outlet port connected to said housing;

an access port connected to said housing; and

a bundle of asymmetric, microporous, hollow fiber membranes disposed within said interior chamber, said asymmetric, microporous, hollow fiber membranes comprising a polyimide polymer, said asymmetric, microporous, hollow fiber membranes including pores having a pore size range from about 0.005 μm to about 0.2 μm , wherein said asymmetric, microporous, hollow fiber membranes have a flux in excess of about 75.times.10.sup.-5 ml/(min.times.cm.sup.2 .times.mmHg); and

wherein said inlet port, said outlet port and said access port are in fluid communication with said bundle of hollow fiber membranes.

2. The filter of claim 1 wherein said fiber comprises 100 wt. % of said polyimide polymer.

3. The filter of claim 1 wherein said fiber comprises 100 wt. % of said polyimide polymer comprising a polymer having the structure: ##STR5## 10% to 90% of the R groups are ##STR6## and the remaining R groups are ##STR7##

4. The filter of claim 3 wherein said fiber comprises from about 100 wt. % of said polyimide polymer.

5. The filter of claim 1 wherein said polyimide polymer comprises a polymer having the structure:

6. The filter of claim 5 wherein said polyimide polymer comprises from about 100 wt. % of said polyimide polymer.

7. The filter of claim 2 wherein said polyimide polymer comprises a polymer having the structure: wherein 10% to 90% of the R groups are ##STR8## and the remaining R groups are ##STR9##

8. The filter of claim 7 wherein said polyimide polymer comprises from about 100 wt. % of said polyimide polymer. The filter of claim 2 wherein said polyimide polymer comprises a polymer having the structure:

9. The filter of claim 1 wherein said polyimide polymer has a molecular weight of about 40,000 daltons to about 105,000 daltons.

16. A dialysis apparatus comprising:

a dialysis system comprising an artificial kidney operatively connected to said dialysis system, said artificial kidney including a dialysate inlet port;

a female to female connector; and

a dialysate filter, said dialysate filter comprising:

a bundle of asymmetric, microporous, hollow fiber membranes;

a filter housing defining an interior chamber;

a dialysate inlet port connected to said filter housing;

a dialysate outlet port connected to said filter housing;

a dialysate access port connected to said filter housing;

and said bundle of asymmetric, microporous, hollow fiber membranes disposed within said interior chamber, said asymmetric microporous hollow fiber membranes comprising a polyimide polymer, said asymmetric, microporous, hollow fiber membranes including pores having a pore size range from about 0.005 μm to about 0.2 μm , and having a flux in excess of about 75.times.10.sup.-5 ml/(min.times.cm.sup.2 .times.mmHg); said dialysate inlet port, said dialysate outlet port and said access port are in fluid communication with said bundle of hollow fiber membranes; and

wherein the dialysate filter is located upstream of the artificial kidney and is connected through the female to female connector to the inlet port of said artificial kidney.

19. The dialysis apparatus of claim 16 wherein said fiber comprises 100 wt. % of said polyimide polymer.

20. The filter of claim 16 wherein said polyimide polymer comprises a polymer having the structure: ##STR10## wherein 10% to 90% of the R groups are ##STR11## and the remaining R groups are ##STR12##

21. The dialysis apparatus of claim 20 wherein said fiber comprises from about 100 wt. % of said polyimide polymer.

22. The dialysis apparatus of claim 16 wherein said polyimide polymer comprises a polymer having the structure:

23. The dialysis apparatus of claim 22 wherein said polyimide polymer comprises from about 100 wt. % of said polyimide polymer.

24. The dialysis apparatus of claim 16 wherein said polyimide polymer has a molecular weight of about 40,000 daltons to about 105,000 daltons.

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TITLE: Process for the separation of components in an organic liquid medium and a semi-permeable composite membrane therefor

Brief Summary Text (1):

The present invention relates to a process for the separation of components in an organic liquid medium by means of a semi-permeable composite membrane with a porous carrier substrate on which a polymer network obtained by interfacial polymerisation is applied and to a semi-permeable composite membrane therefor.

Brief Summary Text (2):

Semi-permeable composite membranes are known from European patent application 0.311.912. As porous carrier one uses herewith preferably a polysulphone. On the porous carrier is applied by interfacial polymerisation poly-meta-phenylene tetrahydrofuran 2,3,4,5-tetracarboxamide. It is noted that such membranes are suitable for use in aqueous systems, in particular for removing salts from aqueous solutions.

Brief Summary Text (3):

Furthermore it is known from the European patent application 0.275.027 that one may remove by reverse osmosis materials which are dissolved or dispersed in an aqueous solution or dispersing medium wherein the said materials are separated from the solvent respectively dispersing medium. The membranes used herewith are selective permeable for certain components of the mixture to be separated. The here described known processes and membranes are developed for separation processes in water. Herein an aqueous feed solution is brought in contact with a surface of the reversed osmosis membrane under pressure. The water permeability of the membrane is promoted by the applied pressure.

Brief Summary Text (4):

Membranes for reverse osmosis in general may be prepared from a polymer, for example polyamide, as described in the U.S. Pat. No. 4,277,344. From more recent developments appears that thin film composite membranes in particular are suitable for reverse osmosis. Such membranes which have a good salt retention are described in the U.S. Pat. Nos. 4,520,044 and 4,606,943.

Brief Summary Text (5):

The U.S. Pat. No. 4,767,148 describes thin film composite membranes for reverse osmosis, which are manufactured by interfacial polymerisation of a polyfunctional primarily water soluble primary or secondary amine in an aqueous solution with a relatively water insoluble polyfunctional acyl halide in an organic solvent.

Brief Summary Text (6):

Thin film composite membranes in general are prepared by interfacial polymerisation. Mostly one uses reactions of polyfunctional amines with polyfunctional acid halides or polyfunctional isocyanates.

*PVA+AMINE
1 + Phenyl
cyanate*

Kolshew
Aug 16, 1994

Dirt 8/16/1994

Brief Summary Text (7):

According to a known process a porous carrier substrate, in general a polysulphone ultra-filtration membrane is coated with a solution of one of the components whereafter the so coated membrane is brought into contact with a solution of the other reactive component wherein the respective solvents are immiscible.

Brief Summary Text (9):

For ultra-filtration membranes in general the membranes are manufactured by the phase inversion technique. Such membranes also may be used as carrier substrates for the manufacture of thin film composite membranes.

Brief Summary Text (10):

Examples of such membranes are described in the U.S. Pat. Nos. 3,926,748 and 4,039,440.

Brief Summary Text (11):

Sometimes one uses carrier substrates with big pores like microporous polypropylene (Celgard) and membranes prepared by the process of the U.S. Pat. No. 4,798,847.

Brief Summary Text (12):

These composite membranes in general have the same properties as silicone membranes manufactured by solution deposit as described in the U.S. Pat. No. 4,581,043, with this difference that possibly the thickness of the composite layer is thinner whereby productivity may be higher.

Brief Summary Text (13):

Composite membranes with a homogeneous coating are also used for reverse osmosis or ultra-filtration processes for the separation of solvents from hydrocarbons. A fluorine containing silicone coating is described in the U.S. Pat. No. 4,748,288.

Brief Summary Text (14):

An example of such processes for the preparation of membranes for use for the separation of aromates from saturates in amongst others pervaporation can be found in the European patent application 0 312 378.

Brief Summary Text (15):

The use of homopolymers for membrane applications has various disadvantages.

Brief Summary Text (16):

In the first place the choice of polymers is restricted, in particular in the cases where the membrane is in contact with liquids like in reverse osmosis, ultra-filtration and pervaporation. The choice herein is restricted to polymers which do not dissolve or do not excessively swell in the dispersing medium or which have functional groups which are capable of causing cross-links.

Brief Summary Text (17):

So, for example, polyvinylalcohol is only suitable as a pervaporation or reverse osmosis membrane for aqueous applications after cross-links since otherwise the polymer would dissolve.

Brief Summary Text (18):

An other example is the use of polydimethyl siloxane as a membrane for removing lubricating oil (dewaxing) from a mixture of methyl ethyl ketone and toluene. Composite membranes with a polydimethyl siloxane coating show in such medium a too excessive swelling reason they cannot be used. To solve this problem one has to use a fluorized siloxane polymer, for example.

Brief Summary Text (19):

The Japanese patent 6 0032-897 describes a process for dewaxing sunflower oil, rape seed oil, cotton seed oil, maize oil, palm oil, coconut oil using a porous hollow

fibre membrane which preferably is a microfilter. This membrane is no composite membrane as meant in the sense of the present invention.

Brief Summary Text (20):

Furthermore U.S. Pat. No. 4,595,507 describes a process for the separation of a solvent from deasphalted crude oil using a polysulphone membrane sulphonated in solution.

Brief Summary Text (21):

Usually a mixture of crude oil and hexane is led through the membrane having a pore size of 10-500 .ANG..

Brief Summary Text (22):

The U.S. Pat. No. 4,541,972 describes a cellulose-1,7-1,8-acetate membrane for the separation of polar solvents from oils. Herein are considered as solvents (m) ethanol or (iso)propanol and cyclohexanone, propylene carbonate, methylisobutyl ketone, tetrakis, chloro benzene or toluene.

Brief Summary Text (23):

The present invention aims a process for the separation of components in an organic liquid medium, for example toluene dissolved n-docosane using a semi-permeable composite membrane which is applied to a porous carrier substrate.

Brief Summary Text (24):

Herefor the present invention provides a process for the separation of components in an organic liquid medium with a semi-permeable composite membrane with a porous carrier substrate on which a polymer network obtained by interfacial polymerisation is applied characterized in that the organic liquid medium is brought in contact with such semi-permeable composite membrane of which the polymer network is built from a reactive polyfunctional monomer or oligomer or a prepolymer with as reactive groups --NHR.sub.1 (R.sub.1 =H or alkyl with C.sub.1 -C.sub.20), --OH or --SH and one reactive polyfunctional monomer or oligomer or prepolymer with as reactive groups --NCO.

Brief Summary Text (29):

According to the invention good results are obtained when use is made of a semi-permeable composite membrane built from a reactive polyfunctional monomer or oligomer or prepolymer or polymer with as reactive groups --NCO from a poly (butadiene) with terminal --OH groups and an aromatic or aliphatic isocyanate with at least two --NCO groups.

Brief Summary Text (30):

The process of the invention proceeds particularly favourable when use is made of a composite membrane of which on the carrier substrate a polymer network obtained by interfacial polymerisation is applied, which polymer network is built from a polyalkyl siloxane either branched or not with final --OH groups with the formula ##STR2## wherein R.sub.2 and R.sub.3 independently from each other represent a C.sub.1 -C.sub.20 alkyl or aryl group either substituted or not and n is an integer of 2-100 or a copolymer thereof.

Brief Summary Text (31):

Good results are obtained by using a composite membrane of which the polymer network is built from a polyethylene imine and toluene diisocyanate.

Brief Summary Text (32):

Also one obtains good results by using a composite membrane of which the polymer network is built from N,N'-bis(3-aminopropylethylene diamine) and toluene diisocyanate.

Brief Summary Text (33):

Furthermore the invention relates to semi-permeable composite membranes with a porous carrier substrate for use in the process of the invention.

Brief Summary Text (34):

A suitable composite membrane of the invention is one wherein on the carrier substrate a polymer network obtained by interfacial polymerisation is applied which polymer network is built from a reactive polyfunctional monomer or oligomer or prepolymer with as reactive groups --NHR.sub.1 (R.sub.1 =H or alkyl with C.sub.1 - C.sub.20), --OH or --SH and one reactive polyfunctional monomer or oligomer or prepolymer with as reactive groups --NCO.

Brief Summary Text (35):

A suitable semi-permeable composite membrane of the invention is one of which the polymer network is built from a reactive polyfunctional monomer or oligomer or prepolymer or polymer with as reactive groups --NCO from a polyalkylene oxide with final --OH groups either branched or not and either substituted or not and an aromatic or aliphatic isocyanate with at least two --NCO groups wherein the polyalkylene oxide preferably is poly(tetramethylene ether glycol) with the formula HO--(CH.sub.2 CH.sub.2 CH.sub.2 CH.sub.2 --O).sub.n --H wherein n is an integer of .gtoreq.3, preferably .gtoreq.6.

Brief Summary Text (37):

Further a semi-permeable composite membrane is suitable when the reactive polyfunctional monomer or oligomer or prepolymer or polymer with as reactive groups --NCO is built from a poly(butadiene) with terminal --OH groups and an aromatic or aliphatic isocyanate with at least two --NCO groups.

Brief Summary Text (38):

Finally, for the separation of components in an organic liquid medium a semi-permeable composite membrane is effective when on the carrier substrate a polymer network obtained by interfacial polymerisation is applied which polymer network is built from a polyalkyl siloxane with terminal --OH groups either branched or not with the formula ##STR4## wherein R.sub.2 and R.sub.3 independently from each other represent a C.sub.1 -C.sub.20 alkyl or aryl group either substituted or not and n is an integer of 2-100 or a copolymer thereof.

Detailed Description Text (3):

A wet flat support membrane of polyimide (0.35.times.0.12 m) prepared from a 16%, by weight, solution of a polyimide type (Lenzing P84) in DMF was applied to a cylindrical immersion body made from teflon. This support membrane was immersed during 15 minutes in a water phase with 0.5%, by weight, NH.sub.2 --CH.sub.2 CH.sub.2 CH.sub.2 --NH--CH.sub.2 --CH.sub.2 --NH--CH.sub.2 CH.sub.2 CH.sub.2 --NH.sub.2, 0.05%, by weight, poly(vinyl alcohol) (Mowiol 4-88.sup.R Hoechst) and 0.04% by weight, sodium dodecyl sulphate or surfactant. Subsequently, the membrane was removed from the water phase and the excess of aqueous solution at the under side thereof was removed with filtering paper. After a dripping period of about 7 minutes the membrane was transferred to an organic phase comprising toluene with dissolved therein 0.5%, by weight, above-mentioned prepolymer.

Detailed Description Text (4):

The membrane was held in the organic phase for 1 minute. Subsequently, the membrane was dried for 5 minutes at room temperature and thereafter 15 minutes in an air circulation oven at 90.degree. C.

Detailed Description Text (5):

The reverse osmosis properties of this membrane were determined at room temperature at 40 bar in a solution comprising toluene and therein dissolved 1.0%, by weight, n-docosane (molar mass 310.6 dalton) with as result a toluene flux of 54 l/m.sup.2 /h and a retention of docosane of 72%.

Detailed Description Text (7):

In an analogous manner as described in example I a 2.97%, by weight, solution of a prepolymer was prepared from toluene diisocyanate (T-80) and a poly(tetramethylene ether glycol) (Polymeg 650.sup.R, Quaker Oats Co.; OH contents 2.99 mmols/g). Following the procedure of example I a composite membrane was prepared. The water phase contained 1.0%, by weight, of the amine. The organic phase comprised toluene with therein dissolved 0.5%, by weight, of above-mentioned prepolymer.

Detailed Description Text (8):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 38 l/m.sup.2 /h and a n-docosane retention of 85%.

Detailed Description Text (10):

In an analogous manner as described in example I a 2.96%, by weight, solution of a prepolymer was prepared from 9.02 g poly(tetramethylene ether glycol) (Polymeg 1000.sup.R), 2.03 g toluene diisocyanate (T-S0), i.e. molar proportion Polymeg 1000 / T-80=2/3 and 0.09 g [CH.sub.3 (CH.sub.2).sub.10 CO.sub.2].sub.2 Sn [(CH.sub.2).sub.3 CH.sub.3].sub.2 as catalyst. Following the procedure of example I subsequently a composite membrane was prepared. The organic phase comprised toluene with therein dissolved 2.0%, by weight, of above-mentioned prepolymer.

Detailed Description Text (11):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 97 l/m.sup.2 /h and a n-docosane retention of 74%.

Detailed Description Text (13):

One proceeds in the manner described in example III in the preparation of a composite membrane- The water phase contained 1.0%, by weight, of the amine. The organic phase contained 1.0%, by weight, of the prepolymer.

Detailed Description Text (14):

The reverse osmosis properties of this membrane were determined at room temperature at 40 bar in a 1.0%, by weight, solution of n-docosane and n-hexane with as a result n-hexane flux of 23 l/m.sup.2 /h and a n-docosane retention of 83%.

Detailed Description Text (16):

In an analogous manner as described in example I a 2.96%, by weight, solution of a prepolymer was prepared from toluene diisocyanate (T-80) and a poly(tetramethylene ether glycol) (Polymeg 2000.sup.R, Quaker Oats Co.; OH contents 0.79 mmols/g). Following the procedure of example I subsequently a composite membrane was prepared. The water phase contained 0.5% by weight of the amine. The organic phase comprised toluene with therein dissolved 1.0% by weight of above-mentioned prepolymer.

Detailed Description Text (17):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 113 l/m.sup.2 /h and a retention of n-docosane of 68%.

Detailed Description Text (19):

One proceeds in the manner described in example V in the preparation of a composite membrane. The water phase contained 1.0%, by weight, of the amine.

Detailed Description Text (20):

The reverse osmosis properties of this membrane were determined at room temperature at 40 bar in a 1.0%, by weight, solution of n-docosane in n-hexane with as result a n-hexane flux of 28 l/m.sup.2 /h and a n-docosane retention of 85%.

Detailed Description Text (22):

In an analogous manner as described in example I a 3.0%, by weight, solution of a prepolymer was prepared from toluene diisocyanate (T-80) and polypropylene glycol (PPG 1000, Janssen Chimica, Belgium, OH contents 1.89 mmol/g). Following the procedure of example I subsequently a composite membrane was prepared. The organic phase comprised toluene with therein dissolved 1.0%, by weight, of above-mentioned prepolymer.

Detailed Description Text (23):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 40 l/m.^{sup.2} /h and a n-docosane retention of 83%.

Detailed Description Text (25):

In an analogous manner as described in example I a 3.26%, by weight, solution of a prepolymer was prepared from toluene diisocyanate (T-80) and a polybutadiene with final hydroxyl groups (PBD 2000, Poly Sciences; OH contents 1.25 mmol/g). Following the procedure of example I subsequently a composite membrane was prepared. The water phase contained 1.0%, by weight, of the amine. The organic phase comprised toluene with therein dissolved 1.0%, by weight, of above-mentioned prepolymer.

Detailed Description Text (26):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 17 l/m.^{sup.2} /h and a n-docosane retention of 82%.

Detailed Description Text (28):

In an analogous manner as described in example I a prepolymer was prepared from 11.26 g .alpha.,.omega.-bis (3-hydroxypropyl) poly(dimethyl siloxane) (Tegomer 2110.^{sup.R}, Th. Goldschmidt, West Germany; OH contents 1.21 mmol/g), 4.75 g toluene diisocyanate (T-80), i.e. molar proportion Tegomer 2110 / T-80=1/2 and 0.11 g [CH._{sub.3} (CH._{sub.2})._{sub.10} CO._{sub.2}]._{sub.2} Sn[(CH._{sub.2})._{sub.3} CH._{sub.3}]._{sub.2} as catalyst. The toluene was removed from the reaction mixture by means of vacuum distillation. Subsequently dry chloroform was added to the residue so that a 1.36%, by weight, solution of the prepolymer in chloroform was obtained. In an analogous manner as described in example I a composite membrane was prepared. The water phase comprised 1.0%, by weight, of the amine. The organic phase comprised chloroform with therein dissolved 1.36%, by weight, of above-mentioned prepolymer.

Detailed Description Text (29):

The reverse osmosis properties of this membrane were determined at room temperature at 40 bar in a 1.0%, by weight, solution of n-docosane in n-hexane with as result a n-hexane flux of 48 l/m.^{sup.2} /h and a n-docosane retention of 73%.

Detailed Description Text (31):

In an analogous manner as described in example I a composite membrane was prepared wherein in the water phase 0.5%, by weight, H._{sub.2} N--CH._{sub.2} CH._{sub.2} CH._{sub.2} - --NH--CH._{sub.2} CH._{sub.2} --NH--CH._{sub.2} CH._{sub.2} CH._{sub.2} --NH._{sub.2}, 0.05%, by weight, poly(vinyl alcohol) (Mowiol 4-88) and 0.04%, by weight, sodium dodecyl sulphate were used. The organic phase comprised toluene with therein dissolved 0.5%, by weight, toluene diisocyanate (T-80).

Detailed Description Text (32):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 193 l/m.^{sup.2} /h and a n-docosane retention of 52%.

Detailed Description Text (34):

In an analogous manner as described in example I a composite membrane was prepared

wherein in the water phase 0.2%, by weight, polyethylene imine M.W. 50.000-60.000, Aldrich, 0.05%, by weight, poly(vinyl alcohol) (Mowiol 4-88) and 0.04%, by weight, sodium dodecyl sulphate were used. The organic phase comprised toluene with therein dissolved 0.5%, by weight, toluene diisocyanate (T-80).

Detailed Description Text (35):

The reverse osmosis properties of this membrane were determined in an analogous manner as in example I with as result a toluene flux of 38 l/m.sup.2 /h and a n-docosane retention of 63%.

Other Reference Publication (1):

Journal of Membrane Science, vol. 32, No. 2-3, Jul. 1987 (Amsterdam, NL), G. R. Bartels et al.: Structure-performance relationships of composite membranes: porous support densification.

CLAIMS:

1. A process for the separation of components in an organic liquid medium with a semi-permeable composite membrane with a porous carrier substrate on which a polymer network obtained by interfacial polymerization is applied, characterized in that the organic liquid medium is brought into contact with such semi-permeable composite membrane of which the polymer network is built from a reactive polyfunctional monomer having as reactive groups --NHR.sub.1, wherein R.sub.1 is hydrogen or a C.sub.1 -C.sub.20 alkyl group, --OH or --SH and one reactive polyfunctional monomer or oligomer or prepolymer or polymer having reactive --NCO groups built from a polyalkylene oxide with terminal --OH groups, either branched or not and either substituted or not, and an aromatic or aliphatic isocyanate with at least two --NCO groups.

4. A semi-permeable composite membrane with a porous substrate characterized in that a polymer network obtained by interfacial polymerization is applied on the carrier substrate, said polymer network being built from a reactive polyfunctional monomer having as reactive groups --NHR, wherein R.sub.1 is H or a C.sub.1 -C.sub.20 alkyl groups, --OH or --SH and a reactive polyfunctional monomer or oligomer or prepolymer or polymer having --NCO reactive groups built from a polyalkylene oxide with terminal --OH groups, either branched or not and either substituted or not, and an aromatic or aliphatic isocyanate with at least two --NCO groups.

5. A semi-permeable composite membranes of claim 4, characterized in that the polyalkylene oxide is poly(tetramethylene ether glycol) with the formula HO-(CH.sub.2 CH.sub.2 CH.sub.2 CH.sub.2 --O).sub.n --H wherein n is an integer of .gtoreq.3, preferably .gtoreq.6.

6. A semi-permeable composite membrane of claim 4, characterized in that the polyalkylene oxide is polypropylene glycol with the formula ##STR6## wherein n is an integer of .gtoreq.3, preferably .gtoreq.15.

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<u>L31</u>	L30 and polyimide	3	<u>L31</u>
<u>L30</u>	polyethylene imine same water same alcohol and membrane	35	<u>L30</u>
<u>L29</u>	L28 and polyimide	0	<u>L29</u>
<u>L28</u>	diamino hexane and membrane	26	<u>L28</u>
<u>L27</u>	diaminohexane and membrane	0	<u>L27</u>
<u>L26</u>	polyimide membrane and diamino hexane	0	<u>L26</u>
<u>L25</u>	diamino hexano and polyimide	0	<u>L25</u>
<u>L24</u>	210/500.38 and polyimide same support and amino and groups and crosslinking same heating	5	<u>L24</u>
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<u>L22</u>	L20 and semipermeable	5	<u>L22</u>
<u>L21</u>	L20 and amino gropup	0	<u>L21</u>
<u>L20</u>	L19 and l15	23	<u>L20</u>

<u>L19</u>	L17 and water and alcohol	23	<u>L19</u>
<u>L18</u>	L17 and covalent same bond	0	<u>L18</u>
<u>L17</u>	L16 and heating	29	<u>L17</u>
<u>L16</u>	L15 and l1	44	<u>L16</u>
<u>L15</u>	polyimide and amino	11953	<u>L15</u>
<u>L14</u>	polyimide and amino same fuctional groups	0	<u>L14</u>
<u>L13</u>	polyimide and amino fuctional groups and heating	0	<u>L13</u>
<u>L12</u>	polyimide and aminofuctional groups and heating	0	<u>L12</u>
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<u>L10</u>	5433852	3	<u>L10</u>
<u>L9</u>	l5 and amino groups	7	<u>L9</u>
<u>L8</u>	l5 and functionalized	0	<u>L8</u>
<u>L7</u>	L5 and coating and amina	0	<u>L7</u>
<u>L6</u>	L5 and coating same amina	0	<u>L6</u>
<u>L5</u>	L1 and polyimide same membrane	92	<u>L5</u>
<u>L4</u>	L3 and polyimide membrane	0	<u>L4</u>
<u>L3</u>	L2 and water and amine and alcohols and crosslinking same heating	0	<u>L3</u>
<u>L2</u>	L1 and polyimide membrane and amino group and heating	1	<u>L2</u>
<u>L1</u>	210/500.39.ccls.	147	<u>L1</u>

END OF SEARCH HISTORY